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MONAZITE AND MONAZITE MINING IN THE CAROLINAS*

BY JOSEPH HYDE PRATT AND DOUGLAS B. STERRETT

Introduction

Monazite is one of the minerals which for a long time was considered rather rare in its occurence, but upon a commercial demand arising for it prospectors and engineers soon located large deposits of it in the Carolinas and Brazil, and the supply has always been able to meet the demand. During the past year further sources of supply of monazite have been discovered and developed in Idaho. North and South Carolina, however, are the only states that have thus far put any monazite on the market.

This mineral is essentially an anhydrous phosphate of the rare earth metals, cerium, lanthanum, and didymium (Ce, La, Di) PO_4. There is nearly always present a varying but small percentage of thoria (ThO_2) and silicic acid (SiO_2), which are very probably united in the form of a thorium silicate ($ThSiO_4$). Some monazites contain but a fraction of a per cent of thoria, while others have been recorded that showed the presence of 18 to 32 per cent;

*Paper read at the Chattanooga meeting of the American Institute of Mining Engineers, October 1908.

but the majority contain from 3 to 9 per cent of this oxide. It is the presence of the thorium oxide that gives the monazite its commercial value. The analysis occasionally shows also the presence of other constituents, as the yttrium and erbium oxides, zirconia, alumina, magnesia, lime, iron oxides, manganese oxide, and titanium ozide.

Monazite is light yellow, honey yellow, reddish, brownish, or greenish yellow in color, with a resinous to vitreous luster, and is translucent to subtransparent. It is brittle with a conchoidal to uneven fracture, and is from 5 to 5.5 in hardness. It crystallizes in the monoclinic system, and some crystals have been observed that were 2 inches in length. The more perfect crystals are, however, very small, ranging from an eighth to a sixteenth of an inch in length down to microscopic ones.

The mineral is usually readily recognized after a few samples have been examined. Its color, usually yellowish inclined to reddish, its hardness 5 to 5.5, being readily scratched by feldspar (hardness 6) or quartz (hardness 7), and its high specific gravity, 4.64 to 5.3, are the chief microscopic properties that will aid in distinguishing it. The principal chemical and blow-pipe reactions that can be readily employed to identify monazite are the following: It is incompletely soluble in hydrochloric acid, but is completely and readily acted upon by sulphuric acid. If oxalic acid is added to the very dilute filtered sulphuric acid solution, or to the solution obtained by fusing the mineral with soda, a precipitate is obtained which upon ignition becomes brick red, due to cerium oxide. Before the blowpipe the mineral turns gray, but is infusible. If heated with sulphuric acid, it colors the flame bluish green, due to phosphoric acid.

The presence of the thoria content of the monazite, which is the substance for which the mineral is mined, varies quite widely from .01 to over 7 per cent. The following analyses of thoria will illustrate the variation in the percentage of this oxide.

Percentage of thoria (ThO_2) in North Carolina monazite sand.

	1	2	3	4	5	6	7	8	9	10
ThO_2...	2.15	2.25	6.54	1.27	6.30	2.48	5.87	6.26	3.98	1.93

1. White Bank gold mine, Burke County.
2. Hall Creek, Burke County.
3. Linebacher place, Silver Creek, Burke County.
4. Long Branch, McDowell County.
5. Alexander Branch, McDowell County.
6. MacLewrath Branch, McDowell County.
7. Proctor farm, near Bellwood, Cleveland County.
8. Wade McCurd farm, Carpenters Knob, Cleveland County.
9. Davis mine, near Mooresboro, Cleveland County.
10. Henrietta, Rutherford County.

These results are for the concentrated sand, but in a number of cases they could have been concentrated to a higher degree of purity and thus contain a higher percentage of thoria.

GEOGRAPHY

Monazite is of wide spread occurrence in the United States, though commercial deposits have been found in but few regions. The area in which monazite deposits of commercial value have been found in the Carolinas lies in the south central part of western North Carolina and in the extreme northwestern part of South Carolina. This area covers about 3,500 square miles and includes part or all of Alexander, Iredell, Caldwell, Catawba, Burke, McDowell, Gaston, Lincoln, Cleveland, Rutherford, and Polk counties in North Carolina; and Cherokee, Laurens, Spartanburg, Greenville, Pickens, Anderson, Oconee counties in South Carolina. The larger towns within or near the monazite region in North Carolina are Statesville, Hickory, and Shelby; and in South Carolina, Gaffney, Spartanburg, and Greenville. This monazite region is crossed by the Southern, the Seaboard Air Line, and the Carolina & North Western railroads.

Several deposists of monazite have been located in northeastern Georgia, though their value has not yet been determined. One of these in Rabun County showed a good quantity of both gold and monazite in a preliminary test. In the adjoining Jackson County

of North Carolina, monazite was found in several pannings that were made in the Horse Cove region two miles east of Highlands. At a number of other places in the mountain region of North Carolina monazite occurs in pegmatized gneisses and schists. Several small deposits of fairly rich monazite bearing gravels are reported by Mr. Geo. L. English to occur in Clay County, North Carolina. The lack of large areas of bottom lands, however, limits the value of these deposits. It has also been found to a limited extent in Cub Creek near Wilkesboro, Wilkes county, North Carolina.

PHYSIOGRAPHY

Physiographically, North and South Carolina are divided into three parts. These are the coastal plain, extending from the Atlantic Ocean northwestward for a 100 to 150 miles; the Piedmont Plateau, extending from the limits of the coastal plain northwestward for 100 to 130 miles to the foot of the Blue Ridge; and the mountain region extending northwestward from the Piedmont Plateau to the State lines. The coastal plain and the Piedmont Plateau are prominent in both States, but only North Carolina contains a large portion of the mountain area.

The coastal plain is a broad, nearly flat stretch of country rising from sea level on the southeast to an elevation of a few hundred feet on the northwest, in which direction it is practically limited by the boundaries of the rock formations of which it is composed. The Piedmont Plateau is an elevated district rising from a few hundred feet above sea level on the southeast to 1200 or 1500 feet on the northwest. It forms a plateau much dissected by valleys from 50 to 200 or 300 feet deep, and its regularity is further disturbed by scattered mountain peaks and smaller hills rising above its general level. The features of the plateau are best observed from a prominent ridge or one of the smaller hills of the region. In the mountain region are included the Blue Ridge and its foothills, and the higher mountains to the northwest. The country in the mountain region is exceedingly rough, and the elevations range from 1500 to over 6500 feet.

The region in which valuable deposits of monazite have been found may be defined as a belt from 20 to 30 miles wide and over

150 miles long. This belt lies wholly within the Piedmont Plateau and borders closely on the Blue Ridge, to whose general course it is roughly parallel.

GEOLOGY

Formations

The rocks of the Carolinas monazite region are principally gneisses and schists. These include the Carolina and Roan gneisses; granite gneiss and porphyritic granite gneiss. Among other rocks are massive granite, pegmatite, peridotite and allied rocks, quartz diorite, and diabase.

The Carolina gneiss is of Archaean age and is the oldest and most important rock of the region. It is composed of several types of gneisses and schists which exhibit various degrees of metamorphism. The most common types are mica, garnet, cyanite, and graphite gneisses and schists or combinations of two or more of these types. The mica of the micaceous types may be either biotite or muscovite or both. More or less mica is generally present in all of the types of the Carolina gneiss, while the garnet and cyanite types with or without the graphite type also occur together. The different types of the Carolina gneiss vary in color from light gray to dark gray and are sometimes bluish gray or bluish black where graphite is abundant in them. Some types of the Carolina gneiss are fine grained so that the component minerals are distinguished with difficulty, while others are more coarsely crystallized. Some of the common constituent minerals of the Carolina gneiss are biotite, muscovite, quartz, garnet, cyanite, feldspar, and graphite. The presence of much pegmatitic material is a characteristic feature of much of the Carolina gneiss.

The Roan gneiss is the next oldest formation of the monazite region and is also of Archaean age. It consists of hornblende gneiss and schist, with occasionally the less metamorphosed phase diorite. The hornblende gneisses and schists are composed chiefly of small interwoven and matted hornblende crystals and grade into diorite which contains a noticeable amount of feldspar and has a granitoid texture. The hornblende rocks vary from black to dark green in color. Bands of mica gneiss and schist, possibly of the Carolina gneiss, are included in both large and small masses of the Roan gneiss.

The age of many of the granites and granite gneisses has not been determined though a part are probably Archaean. The granites and their different phases are next to the Carolina gneiss in importance, and are particularly prominent in areas where rich deposits of monazite exist. The types found in the monazite region are biotite granite, muscovite, and hornblende granite, while in some places considerable secondary garnet has developed in the gneissoid granites. The texture of the granites are gneissic or schistose, porphyritic, and massive. Where the granite is both porphyritic and schistose the feldspar phenocrysts often have an augen form, caused by crushing and shearing. Many of the granite masses have much quartz in veins and veinlets throughout their mass. Some of this quartz is massive crystalline and other occurs with more or less well-defined crystal form, or drusy surfaces. The occurrence of quartz veins is not always confined to the granite masses, but in many places extends some distance from the contact of the granite into adjacent formations. The composition of the granite masses near the contact with other formations has in many cases been altered by the partial or complete absorption of inclusions of those formations. This phenomena is particularly evident where a mica granite, by intrusion into a mass of Roan gneiss, has become a hornblende granite near its borders through the absorption of hornblende.

Pegmatite is a common rock throughout the monazite region and is especially prominent in those areas rich in monazite. Two principal methods of occurrence are here recognized. In one the pegmatite occurs in distinct masses or bodies composed of quartz and feldspar, with or without mica and other accessory constituents. The texture of these masses is, in some cases, extremely coarse with the minerals composing the pegmatite separated out in crystals or masses many inches across. The other type is pegmatized gneiss, representing the addition of the pegmatite minerals to the gneiss, with perhaps some recrystallization of portions of the inclosing rocks. The nature of this pegmatized rock varies considerably. In some places secondary quartz is the principal mineral added, while feldspar is present in smaller quantities. In others feldspar is more prominent. Mica may or may not be

present in the pegmatitic material but has generally been plentifully developed in the mass of the gneiss by metamorphism. The feldspar of pegmatized gneisses often assumes a porphyritic form producing augen gneisses. The gneisses and schists are often banded with or cut at all angles by streaks of pegmatitic or granitic material. The recrystallization of the gneisses and schists, with the development of pegmatitic material or the injection of such material through the rocks, may be called pegmatization. In many places the process has proceeded so far that it is very difficult to distinguish pegmatized gneiss from granite gneiss, especially from porphyritic and flow-banded granite gneiss. This difficulty is partly due to the fact that granite and pegmatite are composed of the same minerals and have no sharp division line between the size of their grains.

The peridotites and allied basic rocks are dark-green to greenish black in color and contain one or more of the ferromagnesian minerals, olivine, pyroxene, and hornblende as chief constituents. So far as known these rocks are of Archaean age and are probably genetically connected with the Roan gneiss. Though a relatively unimportant rock of the monazite region, these basic rocks generally outcrop prominently wherever they occur, and many of the outcrops are marked by large rounded "nigger-head" bowlders. The peridotites and allied rocks are often altered to talcose or chloritic soapstone or serpentine. In some cases this alteration is only superficial, but in others whole masses have been so metamorphosed. These rocks generally occur in lens-shaped bodies parallel. or nearly so, to the schistosity of the inclosing rocks.

Quartz diorite of undetermined age is one of the less important intrusive rocks of the monazite region. It is a hard, fine grained rock, composed of granular quartz and feldspar with varying quantities of hornblende. Locally, garnet is distributed promiscuously through it. Quartz diorite occurs in small dikes, from a few inches to several feet thick. cutting the formations at various angles. Their size is offset by their abundance in some sections and resistance to erosion, owing to which they leave much debris over their outcrops in the form of hard rounded bowlders.

Diabase, probably of Triassic age, is the latest intrusive rock known in the monazite region. It is a dense, hard rock of dark

green to black color, composed chiefly of olivine and a lime feldspar and is rather abundant in some sections and occurs in dikes from a few to over a 100 feet wide. The outcrop is generally marked by abundant characteristic spheroidal "nigger-head" bowlders. The diabase dikes cut the rocks at various angles, though in many cases they have a north to northwest strike.

STRUCTURE

The rocks of this region have undergone extreme regional metamorphism, with accompanying folding and faulting. The mashing and recrystallization of the rocks of the Carolina gneiss formation have been so extensive, in some cases, that much of the original sedimentary structure and igneous texture have been destroyed. The folding of the older formations has resulted, in some places, in complex structure of both large and small dimensions. Some of the folds extend over miles of region, while others are confined to a few feet or inches. The minor deformations and crumplings—miniature Appalachian folds—seen in some rock exposures portray the form of the larger folds. The Carolina gneiss has been intruded by rocks of later age and cut by them into irregular-shaped masses, many of which fork out into long tongues or occur as narrow streaks in the intrusives or vice versa. There have been successive intrusions of igneous rocks of later age into the earlier formations. Thus the Carolina gneiss is cut by the Roan gneiss, and both are cut by grañites of later age.

The structure of the pegmatite in this region is quite variable. In some places the pegmatite occurs in sheets or lenses interbedded and folded with the inclosing gneisses and schists. In other places it occurs in dikes, veins, or lenses either conformable with the inclosing rocks through part of its extent and cutting across them in other parts, or in irregular masses having no definite orientation in the surrounding formations. In pegmatized rock masses pegmatization has generally affected certain beds, which grade into regular pegmatite in either the direction of their greatest or that of their least extension. In such rocks it is often impossible to determine the line of demarcation between the two. There is also a gradation between the pegmatized beds and ordinary gneiss.

ROCKS AND SOILS

The rocks of the southern Appalachain region have undergone extensive weathering and in many places in the Piedmont Plateau, especially, are concealed by a thick mantle of residual soil. In many sections good outcrops are scarce and are found mostly on steep hillsides, along water courses and in road cuts. The residual soils often furnish evidence of the nature of underlying rocks and can be used as a guide to their determination. It is first necessary to learn the different stages of soil formation by the examination of many outcrops and their graduations into residual soil.

The Carolina gneiss, on partial disintegration and decomposition, commonly forms a gravelly soil with a red clayey matrix. This is especially characteristic of the garnetiferous and graphite-cyanite types, which are abundant in parts of the monazite region. The pebbles are composed of small fragments of the original rock, such as tufts of cyanite impregnated with hematite or limonite, iron stained garnets, or pieces of hematite. On more complete decomposition a fine reddish clayey soil results, with no decided characteristics. Other types of the Carolina gneiss, in which mica is an important constituent, leaves a micaceous soil, much of which assumes a purplish color. Granite and its various phases, on partial disintegration and decomposition, yields light sandy soils. On more complete decomposition the granites yield soils of a light to dark reddish color, depending on the quantity of ferromagnesian minerals. as biotite or hornblende, in the original rock. The quartz grains of the granite remain as sand mixed through a clayey matrix. This quartz sand is almost everywhere to be seen at the immediate surface, from which the clays have been washed by rains. Where Carolina gneiss and granite are intimately associated, or where pegmatization has been extensive in a body of Carolina gneiss, there results a sandy soil, characteristic of granite, through which are scattered pebbles of hematite and ferruginous cyanite, characteristic of the Carolina gneiss. The relative importance of pebbles in such soils decreases as the quantity of pegmatite or of granite in the rock formations increases. These features of the soils are especially marked on the broad, flat ridges charac-

terizing much of the Piedmont Plateau region. The Roan gneiss leaves a greenish sandy soil on disintegration, and an ocher-yellow to dark reddish brown or chocolate-colored clayey soil on decomposition. Black stains of manganese are associated with many of the soils derived from hornblendic rocks.

A clew to the nature of the rock formations in a given region is often furnished by the character of the gravels in the bottom lands and streams draining that region. Thus in this area a very light colored gravel with much quartz debris indicates a granite or its contact or a very highly pegmatized country rock. Garnets and hematite iron ore, with which blocks of mica or cyanite gneiss are associated, indicate Carolina gneiss. Quantities of black sands in the stream gravels, containing magnetite, ilmenite, hornblende, etc., are characteristic of the Roan gneiss.

Occurrence

Monazite has been found in several varieties of rocks, in the soils derived from monazite bearing formations, and in gravel beds formed through the erosion of these formations. Only gravel deposits have been profitably worked for monazite on an extensive scale, though in some places the surface soils adjoining rich deposits of monazite, or the saprolite or rotted rock underlying them, are found to be sufficiently rich in monazite to be sluiced down and washed.

The percentage of monazite in both the original rock matrix and in the gravel deposits is small, and probably does not often run over 1 per cent. Figures are not available for the percentage of monazite in gravel deposits. From the saprolite underlying the F. K. McCurd mine, three-fourths of a mile northeast of Carpenter Knob, N. C., Mr. George L. English obtained about one-third of a pound of monazite per ton, or about 0.016 per cent. At the British Monazite mine, 3 miles northeast of Shelby, N. C., the quantity of monazite in the hard rock formations was found by Mr. Hugh Stewart, engineer in charge, to run from between 0.03 per cent and less up to over 1.10 per cent.

MONAZITE-BEARING ROCK

Monazite has been observed in the Carolinas in several types of rock, among which are gneiss, pegmatized gneiss and schist, pegmatite, and different varieties of granite. The occurrence of monazite in ordinary pegmatite masses is in large masses of crystals. These have been found varying from an ounce or two to several pounds in weight in the mica mines of Mitchell and Madison counties, N. C.

Most of the pegmatized gneiss bodies which are rich in monazite represent phases of the Carolina gneiss in which the original nature of the rock has been largely obliterated as a result of the addition of new minerals and the recrystallization of the original ones into pegmatitic material. The texture developed during this pegmatization is in many cases porphyritic, in which the feldspar phenocrysts assume somewhat of an augen form. The feldspar phenocrysts range in size from some smallér than a grain of wheat to others the size of a walnut. The porphyritic gneiss may grade into less or more highly pegmatized gneiss, and from the latter into regular pegmatite. This gradation may be between two separate beds or from one part to another of the same bed. In those beds or portions of beds where there has been little pegmatization monazite occurs sparingly. The same is true where pegmatization has been complete and but little of the original gneiss remains. It is, then, the beds of gneissic rock which are rich in secondary quartz and contain numerous small masses of feldspar throughout that cárry the most monazite. In such rocks there is generally much biotite, with graphite and perhaps some muscovite and other accessory minerals, as well as abundant quartz and feldspar. The quartz occurs in layers or scattered grains throughout the rock, inclosing and replacing the other constituents. The feldspar crystals chiefly replace, though they partly displace the other minerals of the rock. Monazite in a rock matrix almost invariably possesses crystal form, often with brilliant faces.

A typical example of rich monazite-bearing rock could be described as follows: The chief constituents of the rock are quartz, feldspar (mostly the potash variety), biotite, graphite, muscovite, monazite, and a little zircon. It has a banded stuc-

ture caused by the more or less separate occurrence of certain minerals arranged in parallel streaks, with a roughly parallel orientation of the crystals or grains of each mineral. The principal features of the banding consist of larger quartz streaks with several smaller ones and individual grains in a regular biotite schist. The other minerals occupy various positions and show diverse relations to the minerals of these bands and to each other. The feldspar is porphyritic and occurs chiefly in individual crystals, some of which are of considerable size. A number of the feldspar phenocrysts are small bodies of pegmatite in themselves. The feldspar phenocrysts replace the other minerals. Graphite occurs in large amounts with biotite, though it is associated with nearly every other mineral of the rock. Where present, muscovite is chiefly associated with the feldspar. Monazite seems to be indiscriminately scattered through the rock, included in or associated with all the foregoing minerals. Though generally free from inclusions, it is not invariably so, and in one case a plate of graphite was observed within a monazite crystal. All the minerals observed in the rock, with the exception of zircon, have been noted as inclusions in the feldspar phenocrysts.

In microscopic sections cut from specimens from one of the ore streaks, the minerals described above were observed, together with some iron staining. The feldspar is principally orthoclase and microcline, partially kaolinized. The quartz is plainly secondary, and occurs in bands or streaks or grains parallel with the schistosity of the rock. In some places the quartz has been deposited in the fracture or between the grains of other minerals; in others it replaces or includes fragments of such minerals as biotite and graphite.

Gas cavities and inclusions of very fine acicular needles, probably rutile, are abundant in the quartz. Biotite occurs in interwoven laths and crystals roughly parallel to the banding of the rock. The pleochroism of the biotite is light yellow-brown to greenish brown or dark purplish red. Graphite occurs as plates and laths, in general lying parallel to the banding of the rock. Some of it is interbanded and even interleaved with biotite; elsewhere the plates are turned across the foliation. In one section a lath of graphite was observed inclosed in quartz which filled a

fracture across the foliation of a biotite crystal. Monazite occurs in contact with the various minerals of the sectoin, though it is more commonly surrounded by or included in grains of biotite and quartz. The position of the monazite in the biotite indicates replacement, and the biotite foliae are not displaced around the crystals. In the microscopic sections sufficient feldspar was not observed to determine its relation to the other minerals.

The rock has been so thoroughly recrystallized that it is difficult to give the relative order of formation of the minerals. Biotite, if not still in its original condition, was probably the first mineral to form during recrystallization. Part of the graphite was probably contemporaneous with the biotite. Some, however, was introduced later and formed at the same time with the quartz. The small amount of muscovite in the rock was probably next to form, followed closely by quartz. From the small amount of feldspar in the microscopic sections, it was not possible to state its relative period of formation. From the hand specimen, however, it is evident that the feldspar was introduced later than the quartz, or possibly contemporaneously with part of it.

ORIGIN

The occurrence of monazite in granitic and pegmatitic rocks indicates that its origin is associated with magmatic agencies. It is probable that the constituents of monazite are associated with granitic magmas and that only part of the mineral crystallizes out when such magmas solidify. During the formation of pegmatite magmas and solutions from the residues of the solidification of granite part of the constituents of monazite are retained. When these pegmatite magmas and solutions are intruded into or deposited in the gneisses and schists in masses such as are mined for mica, monazite forms in large masses or crystals. During the pegmatization of rock formations by these magmas and solutions the monazite is carried into the gneisses and schists where it is now found. This pegmatization with which monazite is associated was probably produced by the passage of active magmatic solutions through the rock, both aiding in recrystallization of the original constituents, and depositing the materials held in solution when conditions of temperature or agents of precipitation were favorable.

It is possible that in some cases the monazite in pegmati gneiss is formed by the gathering together of the proper eleme disseminated through the original rock during recrystallizati It is probable that pegmatization in which much quartz with little feldspar has formed represents a phase of recrystallizat in which the quartz may either in part or wholly have come fr the original rock itself or may have been added by solutions pa ing through the formations. In either case the materials do represent the work of active magmatic solutions or magmas st as might give rise to regular pegmatite bodies. In those recryst lized or pegmatized rocks where the feldspathic component of p matite is not plentiful, monazite occurs but sparingly. On other hand, monazite is found more abundantly in pegmati rock formations in which feldspar plays a prominent part. T common proximity of this form of pegmatization to gran masses, or its gradation into pegmatite bodies, gives evidence its formation through magmatic agencies.

The monazite of rock formations has, then, probably be derived from aqueo-igneous solutions such as give rise to certa forms of pegmatite and have in these cases affected large masses rock.

PLACERS

The commercial deposits of monazite occur in the gravel beds creeks and streams and in the bottom lands adjacent to the The thickness of the gravels ranges from a foot or two, includi over-burden, to 6 or 8 feet. The distribution of the monazite them is, as with all heavy minerals, richer near the bed rock a poorer above, grading into the over-burden. In some deposits t whole thickness of the gravel, with the finer alluvium at the su face, is rich enough to be washed directly or sluiced down an washed. The extent and value of these deposits vary with the topo raphy of the country and the nature of the gravels. In some plac the bottom land, containing rich monazite-bearing gravels, are ov 100 yards wide and extend a half a mile or more along the stream In other places the bottom lands are small and there is but litt more than the stream gravels present. The best deposits are mo commonly associated with light colored gravels and sands, co

taining considerable quartz debris and fragments of other light colored rocks, such as pegmatite, granite, mica, and cyanite gneiss. On the other hand, the absence of much quartz and pegmatitic or granitic debris from the gravel is generally characteristic of low grade deposits of monazite. The presence of black sands—magnetite, ilmenite, hornblends, etc.—in the gravels does not necessarily indicate a low grade deposit, unless quartz and pegmatitic minerals are also lacking. Monazite deposits in regions where hornblende rocks are abundant generally contain a large percentage of black sands, and it is then often difficult to concentrate the monazite to a marketable grade. As an offset to this, however, especially in regions where granite is associated with the hornblendic rocks, gold is often found in the concentrates in quantity more than sufficient to pay the cost of separation, and in the same localities the concentrates generally carry also a quantity of zircon. This zircon is in the form of small, clear crystals with brilliant lustre, which range in size up to 1 millimeter square and about 2 millimeters long.

RESIDUAL DEPOSITS

It has been found profitable to sluice down and concentrate the surface soils on the lands adjoining some of the richer monazite bearing deposits. The residual soils that have suffered but little displacement on the surface can be thus profitably washed to a depth of 3 or 4 inches, and where the drift soil has collected on the gentle slopes below a steeper hillside several feet can be sluiced down in some cases. The partial concentration of monazite in the top layer of soil is caused by the washing away of the clay and other light decomposition products of the rock. The supply of monazite in the stream gravels in favorable areas is often replenished by the wash from the hillside soils during rains; especially where the hills have any considerable slope and the land is cultivated. Under such conditions the stream gravels are often worked two or more times in a year.

The saprolite or rotted rock underlying the richer deposits of monazite is at some places sluiced down to depths of a few inches to a foot or so, along with the overlying gravels. At other places small amounts are removed and washed separately for the mona-

zite they contain. The formations that have been found espe
ally favorable for such work are highly pegmatized gneiss or sch
Such deposits have generally soon been lost or grown poor, pr
ably on account of the fact that the miners have cut through
richer bed or failed to follow it in the direction of its extensi
The occurrence of monazite in saprolite is merely an altered ph
of the occurrence in hard rock formations.

COMMERCIAL DEPOSITS

All the monazite mined in the Carolinas is obtained from gra
deposits which lie in and along the stream and creek beds wh
the monazite is collected after being liberated from the rocks
their alteration and erosion. While no accurate record has be
kept of the percentage of monazite in these gravel deposits, yet
is undoubtedly true that the percentage per cubic yard, reckoni
from surface to bed rock, is not over 1 percent. This, howev
is sufficient to make profitable mining. In many localities it h
been the custom to sluice not only the gravels but all the ov
burden, inasmuch as even the top soil carries a small amount
monazite.

There are no large hydraulic plants in operation, but nearly
the monazite is obtained in sluice boxes fed by hand. The
boxes are fitted at their upper end with a sieve or shaking hopp
with a mesh of about No. 12. The boxes vary in length from
to 20 feet, and in some instances are fitted with riffles holdi
mercury for catching the gold. An interesting fact noted in co
nection with the deposition of monazite in the stream beds is th
when the gravels have been washed for monazite and then left fo
few months or a year (especially if there has been considerable rai
weather), there is another supply of monazite deposited, which
many cases can be profitably worked. This monazite has result
from the washing in of the mineral from the surface adjoining t
streams where it had been during the decomposition and erosi
of the original rock matrix. This second deposition of monazi
is facilitated by plowing the adjoining fields. In a few plac
Wilfley tables have been introduced for treating the concentrat
from the sluice boxes. Where these tables are used the soil ar

gravels are washed into shaking hoppers and then through sluice boxes, the over size thrown out and the sands fed to Wilfley tables. At one mine it is necessary to raise the gravels by a mechanical elevator in order to bring them to a sufficient height to feed them to the table. They are fed into a revolving screen and from that to the table. The heads from this first washing do not contain a very large per cent of the monazite, and the middlings are, therefore, re-fed to the table with other feed ore. In some cases the feed ore is all run over the machine and a rough concentrate first obtained and then this re-fed. The product from these machines contain from 50 to 80 and occasionally 90 per cent of monazite. Where there is a large amount of the heavy black sands occurring in the gravel with the monazite, it is almost impossible to get the percentage much over 50 per cent monazite. Where, however, these occur more sparingly, it is possible by this method to obtain a monazite concentrate containing 80 per cent monazite.

All the concentrates from the sluice boxes and Wilfley tables have to be dried before they can be treated on the magnetic separators. There are two different methods used in the monazite district for this purpose. In one the sand is spread over an oiled or rubber cloth in a thin layer and exposed to the heat of the sun. It dries very quickly, due perhaps partly to the heat absorbed by the dark iron sand. It requires, however, a considerable surface to accommodate any large amount of sand. The other method of drying is by heating over furnaces. A small ditch from 4 to 8 feet long and 1½ to 2 feet wide and about one foot deep is dug, at one end of which there is built a rock or brick chimney. The ditch is usually built up of stones with an opening at the opposite end of the chimney for firing. Over the ditch there is a sheet iron cover or drying plate. The monazite is spread on this and exposed to the action of the hot fire underneath. These dry sands are often further concentrated by means of the ordinary horseshoe magnet, which picks out all the magnetite. The miners are paid for their sand on the basis of 100 per cent product and the nearer they can bring their sand to this, the better prices they receive for

it. The sand brought into the magnetic concentration plants worth from 4 to 8 cents per pound while after a magnetic sepa tion, its value is increased to 12 to 20 cents per pound.

This material represents what is known as crude monazite sa and contains, besides the monazite, magnetite, ilmenite, garn zircon, rutile, corundum, cyanite, hornblende, and occasiona chromite. In order to separate the monazite from its associat minerals, it is necessary to run this crude sand through some el trical apparatus. There are two types of machines that are operation: (1) the Wetherill electro-magnetic machine and mo fications of this; and (2) machines in which the minerals deflected by electro-magnets while falling. Of these, the first ty is the one most generally employed. By means of these vario machines a product can be obtained varying from 90 to 98 p cent of monazite and represents the sand that is shipped to t manufacturers of the incandescent mantles.

MAGNETIC SEPARATION

The first application of magnetic separation was in the conce tration of certain iron ores, principally magnetite, in order to pr duce a product richer in iron and also to eliminate certain mine als that contained elements injurious to the metallic iron. T next application was to other iron ores such as limonite, hematit and siderite after they had been given a preliminary roasting convert them into the magnetic oxide. The next step was in t separation of magnetic iron particles from certain copper, go and zinc ores either before or after roasting. For many years th was the only application made of magnetic separation. It w found, however, upon experimenting with an electro-magnet wi a higher intensity that other minerals were subject to magnet attraction and that it was possible to separate minerals into mo or less pure products by varying the intensity of the magnet field. Thus, it has been possible to adapt this method of separ tion to ores containing iron or manganese, which are only weak magnetic. As is well known, steel bars may be magnetized a they will retain more or less of this magnetism indefinitely, whi bars of softer wrought or cast iron may be magnetized by mea of electric currents in surrounding coils of insulated copper wir

These iron bars do not become permanent magnets but form electro-magnets as long as the current flows around them. They can be given a greater and more constant strength than can be given to the permanent steel magnets and for this reason, in nearly all of the magnetic processes, electro-magnets are used instead of the field magnets.

The magnetism of these electromagnets can be varied and different intensities obtained ranging from indefinitely weak to a certain maximum strength. It is also possible to control the intensity of any magnetic field so that minerals that are strongly attracted may be separated from minerals that require a magnetic field of much higher intensity. This intensity of the magnetic field depends:

1. On the size of the magnet.
2. On the shape of the magnet.
3. On the distance between the magnet and the body to be attracted.
4. On the number of amperes turns in the magnet coil, that is, the product of the amperes or current flowing in the coil times the number of turns around the core.

There are many substances that are attracted by electro-magnets that are not influenced apparently at all by the strongest steel magnet and for this reason, formerly many substances which were considered non-magnetic, have been proved to be magnetic when subjected to the intense magnetic field obtained in an electromagnetic separator. All substances are of course either attracted or repelled by magnets and the former are called para-magnetic and the latter dia-magnetic. The latter class is the most numerous, but since the introduction of electromagnets, the former class, which up to this time had been considered extremely small, has been largely increased. The paramagnetic substances are the metals iron, nickel, cobalt, manganese, chromium, cerium, palladium, platinum, osmium and many of their salts and compounds. The degree of attraction of these varies very widely and, as an illustration between a strong and weak magnetic substance, it has been estimated that if the attraction of steel be taken at 100,000, then magnetite would be 65,000, siderite 120, hematite, 93 to 43, lim-

onite 72 to 43. By using the electromagnetic separators, wh
can be regulated so as to give a very strong field and at the s
time a field which is capable of fine adjustment, it is now poss
not only to separate the paramagnetic from the diamagnetic s
stances, but also to separate the paramagnetic from each other

There are a large number of magnetic separators that have b
invented, many of which are now on the market. Perhaps
simplest of all these magnetic separators is one devised by Edis
In this separator the particles of mineral are permitted to fall i
thin sheet in front of the poles of a strong bar electromagnet, wh
causes a deflection of the magnetic particles from a direct do
ward path, while the nonmagnetic particles would not
influenced by this attraction and would fall vertically. It is po
ble to make two and sometimes three products in this way.

There are three general classes of these magnetic separators
follows: (1) in which the magnetic particles are held to revolv
cylindrical rolls or drums within which are magnets; (2) th
in which the magnetic particles are carried by conveying belts
pans passing over the magnets; (3) those in which the ore falls
front of a magnet. There are a number of points of difference
the machines such as permanent or electromagnets; treating
ore wet or dry; magnets acting continuously or intermittentl
and the use of direct or alternating current. It will be found th
different machines are suited for different purposes according to
character of the material to be treated. As I have stated befo
most of the machines were originally designed simply to treat ir
ores, or to separate iron minerals from other ores and there
but few of them that are adapted for the separation of monazi
zinc minerals, etc.

The first class is represented by the Ball-Norton separator wh
consists of two revolving drums within each of which is a series
stationary electro-magnets so wound that opposite poles are ad
cent to one another. The capacity of a machine with 2 drums
dia. and 2' face raises 15-20 tons per hour, 16-20 mesh. The
is fed upon the top of the first drum and the magnetic partic
are held by the drum, while the non-magnetic fall into the hopp
below. As the drum revolves, the magnetic particles get beyo

the magnetic field and are thrown by centrifugal force on to the second drum. This drum, which does not have quite so strong a current as the first, does not attract as many of the magnetic particles so that some of these drop off into a second hopper, forming a middling product, while the stronger magnetic particles are held by the drum and carried a certain distance, when they get beyond the magnetic field and are dropped into a third hopper. On account of the alternate polarity of the adjacent magnets, the particles roll over and thus facilitate the elimination of any gangue particles that may be mixed with the magnetic.

Another simple drum separator is the Heberli. In this machine there is but one drum and the electro-magnets extend over about one-fourth of the area of the drum. The ore is fed to the drum just above the centre radius and about the middle of the magnets. The drum revolves in the opposite direction to which the ore is fed and the magnetic particles are attracted by the drum and carried up and over the magnets while the non-magnetic particles drop into the hopper below. As the magnetic particles leave the magnetic field, they are dropped on the opposite side of the drum into another hopper.

2. It is the magnetic separators of the second class that have been used principally in the separation of monazite in the Carolinas. Of these machines, the Wetherill stands out most prominently and was probably the first to commercially treat weakly magnetic materials. The principal idea of these machines is to secure a very strongly magnetic field by concentrating the lines of force as far as possible, this being accomplished by placing the two poles of the magnet facing one another with a minimum air gap between them and by bevelling down the pole pieces to their end.

The type of the Wetherill magnetic separator that is more generally used is known as the Rowand type, which has a magnetic pole with sharp edge between the travelling feed belt and a blunt pole directly under it. Both of these poles are capable of being magnetised by an electric current which will produce a condition varying from weak to intensely strong magnetism. The concentration of magnetism at the sharp edge causes all the grains to jump to the upper pole. A cross-belt directly beneath this pole, which is running at right angles to the feed belt and is running

rapidly, readily takes off these grains and deposits them in a
while the non-magnetic grains go on with the belt. There can
readily arranged above the travelling feed belt a series of s
poles, each stronger than the one before, so that the first will t
off the strongest magnetic particles. The travelling feed
varies in width from 12 to 18 inches. The material fed to
machine is classified and allowed to pour over a revolving dru
which concentrates it evenly over the feed belt. The pole pie
are made of soft iron and weigh up to 90 pounds each. They
adjustable so that the length of ore gap between them may
varied. The strength of the current in amperes can be varied a
also the distance of the feed belt beneath the poles.

The monazite sand, which is fed to the travelling feed b
passes along under four powerful electo-magnets. The first
moves all the magnetic iron and generally all of the titanic iron
ilemnite and any chromite that might be present. The seco
magnet removes all the fine grains of garnet, the coarser ones,
present, usually being removed by the first magnet. The th
magnet is so adjusted as to remove only the coarser particles
monazite, while the fourth removes all the finer pieces of mo
zite. The remaining portion of the sand, consisting largely
zircon, quartz, and a little rutile, corundum, cyanite, etc.,
dropped off at the end of the large belt into a waste pile.

In another type of machine used in the monazite district th
are a series of magnets over which are travelling belts which p
out different minerals, according to the intensity of the magne
field. In this machine the magnetic particles are carried over a
under the magnet and dropped into a hopper as they leave
magnetic field, while the tailings are dropped into another hop
and fed to another travelling belt and over a second magnet
stronger intensity, which picks out the garnet. This is dropp
into a special bin and the balance into another hopper and fed
a third magnet, whick picks out the monazite. It is possible
these separators to obtain a monazite sand of from 90 to 99
cent monazite, according to the care that is taken in separating

The other products, as the iron minerals magnetite and ilmeni
and garnet, can also be obtained in a very pure state. From
long series of experiments that have been carried on, it has be

determined that in machines of this type the magnetite can be removed when the amperage is .2; ilmenite with 1.1; chromite with 1.6; garnet with 1.75; hypersthene and olivine with 2.2; monazite with 3.5 amperes. Zircon is left behind with the gold as non-magnetic. Any platinum that might be present would begin to be lifted by the weakest current, but most of it would not be lifted until the current was 1.5 amperes.

It is possible to separate almost completely pyrite from hornblende by picking out the hornblende with the electro-magnet, the pyrite remaining in the tailings. Such minerals as pyroxene, epidote, titanite, tourmaline, and serpentine are readily picked out by the Wetherill magnetic separator with a current of 2 to 2.5 amperes. Brookite and cassiterite can occasionally be picked out with an amperage of 3.5.

USES OF MONAZITE

The commercial value of monazite depends upon the incandescent properties of the rare earth oxides which it contains, such as cerium, lanthanum, didymium and thorium oxides, which are used in the manufacture of the Welsbach and other incandescent gas light mantles. It is the thoria that is used in largest amount and which gives the actual value to the monazite. In the reduction of the monazite sand, there are a number of the rare earth salts that are obtained in considerable quantity, which has made it possible to carry on an extensive series of experiments with these rare earth oxides. It requires from 4 to 6 months to recover from the monazite sand its percentage of thoria and render it sufficiently pure to be used in the mantles.

The Welsbach light consists of a cylindrical hood or mantle composed of a fibrous network of the rare earths, the top of which is drawn together and held by a loop of asbestos or platinum wire. When in use, this mantle is suspended over the flame of a burner, constructed on the principle of the Bunsen burner, in which the heating instead of the illuminating power of the hydrocarbon of the gas is used by burning it with an excess of air. In this manner the mantle becomes incandescent and glows with a brilliant and uniform light.

A short description of the method of manufacture of these mantles may be of interest. The first part of the process is the selection of the thread fibre from which the mantle fabric is knitted. The fibre mostly used is cotton, either the upland, river bottom, Peeler, Allen seed, Sea Island or Egyptian variety, the market prices varying from about 10c for the upland to 30c per pound for the Egyptian. The cheaper cottons are used in the lower grade mantles, the highest grade mantle requiring the best quality of cotton. The thread is purified, so as to remove every possible trace of mineral matter. If the thread used shows a mineral impurity above .015 per cent, it will introduce factors that will affect the physical and lighting life of the mantle. Cylindrical networks of varying diameters are knitted out of the thread and then washed in ammonia and distilled water and wrung out in mechanical clothes wringers. After it is dry it is cut into pieces sufficiently long to make two good mantles.

These knitted fabrics are then placed in a suitable vessel and covered with the "lighting fluid." They remain in this until thorougly saturated. The excess of fluid is drawn off amd the fabric put through an equalizing machine piece by piece. The "lighting fluid" is composed of a solution of approximately 99 per cent thorium nitrate and 1 per cent cerium nitrate in distilled water, in the ratio of 3 parts of water to 1 part of mixed nitrates. The fabric is dried and then cut to the proper length required for a hood. They are then shaped over a wooden form and the upper end drawn together by means of an asbestos cord (occasionally of platinum). After the mantle has been modelled the cotton fibre is eliminated by heating them over a hot Bunsen burner flame, leaving the mantle composed of the ash of thorium and cerium. The peculiarity of these oxides is that they have sufficient cohesion to hold together during the balance of the process of manufacture, after every bit of the supporting cotton thread has been burned away. They are then subjected to a series of tempering and testing heats during which the mantle is carefully shaped to its permanent form. In order to protect the ash of the mantle during its inspection, packing, transportation, and installation, it is dipped in collodion. Just before using a mantle this collodion cover-

ing has to be burned off. It is estimated that the American market consumes 40,000,000 of these mantles per year.

Another element obtained from the monazite is didymium, whose oxide is dark brown. Use is made of this for branding the mantles with an indelible brand. A nitrate solution is made and an ordinary rubber stamp used for branding.

Of the associated minerals, zircon has a commercial value of 20 to 25 cents per pound for its zirconia content, which is used in the manufacture of the glower of the Nernst lamp. The fundamental principle of this Nernst lamp is that certain of the rare earths or refractory oxides will conduct an electric current and glow after they have been heated to redness. This discovery, which was made by Dr. Nernst in 1897, has resulted in the development and perfecting of the glower which is now embodied in the Nernst lamp. This glower is composed of a mixture of the rare earth oxides and is made in the form of a small rod or pencil of chalk-like material, having wire terminals at either end. When cold, the glower is an insulator, but by means of the wire the glower becames heated to redness when a current is passed through these wires, and its resistance gradually decreases until it has reached a red heat, when with 220 volts across the terminals it starts to conduct the current and give light.

In bringing a glower up to its starting point corresponding to a temperature of 1,200° F., use is made of a small electrical heater composed of two or more small tubes of porcelain, about 1½ inches long and ¼ inch in diameter, which are overwound with fine platinum wire, this in turn being held in place and protected from the intense heat later generated by the glower by an outer coating of porcelain paste. After the glower becomes heated, there is, of course, no further use for the heater, and it is cut out by a small electro-magnet cut-out, which consists of a magnetic coil connected in series with the glower, an armature, and the necessary contacts in the heater circuit. Thus, when the glower has become heated sufficiently, the current begins to pass through it, and when this becomes sufficiently strong the armature is attracted and the contacts are separated, thus disconnecting the heater from the line. The surface of the glower before being used presents a

smooth, white, porcelain or chalky appearance, but after having been in use about 500 hours, it is rough or crystalline in appearance.

The yttria contents used in the manufacture of the Nernst glower are obtained principally from the mineral gadolinite, which has not thus far been found in North Carolina. There are, however, a number of minerals containing yttria, such as samarskite, euxenite and fergusonite, which have been found in the State.

The magnetite and ilmenite may find a use in the manufacture of magnetite electrodes that are manufactured by the General Electric Company.

The garnet grains are sharp and can be used for abrasive purposes in the manufacture of garnet paper, which is used extensively in the boot and shoe trade.

THE OPTICAL ROTATION OF SPIRITS OF TURPENTINE*

BY CHAS. H. HERTY

Among the physical properties of spirits of turpentine, none has proved of more interest than its optical rotation. In most specimens this property is very marked, and as the liquid is colorless and the determination readily made, many data are found on this subject in chemical literature. Slight variations in the rotation of different samples are to be expected, as spirits of turpentine is not a chemical compound but a mixture of substances, chiefly terpenes. From the results of numerous observations upon commercial samples, the view commonly held previous to 1891 was that French spirits of turpentine, distilled from the oleoresin of *Pinus maritima*, is levo-rotatory and that American spirits of turpentine, distilled in years past, almost wholy from *Pinus palustres*, is dextro-rotatory. The difference in the character of the rotation was ascribed, therefore, to the different species from which the spirits of turpentine was produced.

Recognizing the fact that American spirits of turpentine is distilled from more than one species of pine, J. H. Long,[1] in 1891, undertook a study of the volatile oils distilled from oleoresins of well identified individual trees, these trees embracing the several species of pines subjected to turpentining in our southern states. He found that specimens from *Pinus palustris* (Long Leaf Pine) gave dextro-rotatory oils, while those from *Pinus heterophylla* (Cuban or Slash Pine) gave levo-rotatory oils. Since the oleoresins from these two species are indiscriminately mixed, at the time of collection in the woods, the rotation of the oil distilled from such a mixture would naturally vary. *Pinus palustris* is the

*Reprinted from the Journal of the American Chemical Society, vol. 30, p. 863.

[1] *J. Anal. Appl. Chem., 6, 1.*

predominating species and Long attributed to this fact the dextrorotatory character of American spirits of turpentine. This view has been generally accepted.

The fact that spirits of turpentine is frequently adulterated with optically inactive mineral oil, led A. McGill[2] to make a large number of determinations of the rotation of commercial samples of spirits of turpentine, in the hope of utilizing this property for the detection of adulteration. From the widely varying results obtained he was compelled to declare the method useless.

New evidence upon this point has been obtained from investigations carried on in this laboratory in collaboration with the U. S. Forest Service, the experimental work having been carried out by Messrs. George A. Johnston and W. S. Dickson under the direction of the writer. In order to gain a better knowledge of the oleoresins from the two principal species of pine utilized in the turpentine industry at the present time, fourteen trees were selected on a Florida turpentine farm. One-half of these were *Pinus palustris*, the other half *Pinus heterophylla*. Three trees of each species were tapped for the first time at the beginning of the experiments. In each case a small, young pine, a medium pine, and a large, old pine were selected. In another set four trees were selected, two each *Pinus palustris* and *Pinus heterophylla*. These trees had been subjected to turpentining during the previous year, the chipping or weekly scarification, on all of them having been unusually shallow, only about one-half as deep as is commonly practiced. In a third set four trees were selected, two of each of *Pinus palustris* and *Pinus heterophylla*, which had been turpentined during the previous year, and on each of these the depth of the chipping was the normal cut. The trees in each set were chipped at intervals of seven days.

Special precautions were taken in the collection of the oleoresins. The cup and gutter system described in *Bulletin No. 40, U. S. Bureau of Forestry*, was used. Instead of the clay cup commonly used, oyster pails were substituted. The entire apparatus was covered with black oilcloth fastened securely into the bark of the tree above the chipping surface, thereby protecting the resin

[2] *Bulletin No. 79, Inland Revenue Dept., Canada.*

from light and avoiding the filling of the pails with rain water. Every four weeks these pails were removed from the tree, tightly stoppered and immediately shipped to this laboratory for examination. The specimens so obtained were extremely pure and free from chips. After removal of the pails, the metal gutters were raised to a point near the shipping surface in order to minimize the amount of oleoresin which might stick to the exposed portion of the trunk above the gutters.

The distillation of the oleoresins was carried out in a 500 cc. Kjeldahl flask, surrounded by a bath of cottonseed oil. Steam from a small boiler was first passed through a small iron pipe in which it could be superheated, then into the distillation flask through a glass tube having on its end a bulb containing a number of openings. By this means strong agitation of the molten oleoresin was obtained. Thermometers were placed both inside the flask and in the oil-bath. The mixed vapors of steam and spirits of turpentine were passed through a Hopkins condensing bulb to prevent the carrying over of solid particles of resin, condensed in an ordinary Liebig condenser and collected in a separatory funnel. After drawing off the lower layer of water, the spirits of turpentine was transferred to a dry flask and allowed to stand over night with calcium chloride. The determinations of the optical rotation of the volatile oils were made with a Schmidt and Haensch half-shadow polariscope, sodium flame, at 20°.

In the following table are given the results from the first collection of the oleoresin in early spring:

TABLE I

Tree designation	Species	Diameter (inches)	Character of chipping	Optical rotation 100 mm tube, 20 C
A1	*P. heterophylla*.....	7.0	1st year, normal depth	−20°50′
A2	"	14.5	" "	+ 0°15′
A3	"	24.5	" "	−15° 0′
A4	P. *Palustris*.....	7.3	" "	+15°40′
A5	"	15.0	" "	+ 8° 9′
A6	"	21.0	" "	+18°18′
C1	*P. heterophylla*.....	12.3	2nd year, shallow	−27°11′
C2	"	8.2	" "	−26°28′
C3	*P. palustris*.....	13.0	" "	− 7°26′
C4	"	8.7	" "	+ 7°31′
D1	"	9.0	2nd year, normal depth	+10°50′
D2	"	13.5	" "	+ 1°23′
D3	*P. heterophylla*.....	13.0	" "	−18°35′
D4	"	9.0	" "	−29°26′

These results show a wide variation in the optical rotation of the volatile oils from the individual trees, even among trees of the same species. In a general way the figures give support to Long's view, namely that the volatile oils from *Pinus palustris* are dextro-rotatory and those from *Pinus heterophylla* levo-rotatory. That this is not strictly true, however, is evidenced by the dextro-rotation of A_2 (*P. heterophylla*) and more especially by the levo-rotation of C_3 (*P. palustris*).

With these variations in the first collection from the several trees, the question naturally arose, would the variations change as the season advanced or would the figures prove constant for the individual trees? The rotations for the successive collections follow in Table II:

TABLE II.—OPTICAL ROTATION IN 100 MM. TUBE, 20° C.

Collection	A1	A2	A3	A4	A5	A6	C1
1..	−20°50′	+0°15′	−15° 0′	+15°40′	+8° 9′	+18°18′	−27°11′
2..	−22° 5′	−0°30′	−14°26′	+15°22′	+8°50′	+17°43′	−26°48′
3..	−21°45′	+0°15′	−15°55′	+14°15′	+8°27′	+19°30′	−26°25′
4..	−21° 7′	−1°15′	−15°50′	+14°20′	+8°34′	+18°46′	−23°32′
5..	−20°30′	−2° 5′	−15°15′	+14°21′	+8°32′	+19°24′	−21°12′
6..	−20°15′	−3°30′	−15°27′	+14°35′	+8° 4′	+18°16′	−21°46′
7..	−22°15′	−5°45′	−17°52′	+12°49′	+7° 6′	+14°47′	−21°35′

Collection	C2	C3	C4	D1	D2	D3	D4
1..	−26°28′	−7°26′	+ 7°31′	+10°50′	+1°23′	−18°35′	−29°26′
2..	−25°37′	−6°42	+ 7°20′	+11°23′	+2°40′	−17° 0′	−27°45′
3..	−26°20′	−4°45′	.....	+13° 7′	+2°25′	−15°20′	−28°19′
4..	−26°30′	−4°29′	.....	+12°46′	+2°25′	−15° 0′	−27°38′
5..	−26° 7′	−3°55′	.....	+13° 0′	+1°13′	−14°38′	−27°48′
6..	−26° 0′	−4° 5′	.....	+13° 0′	+1°15′	−14° 7′	−26°11′
7..	−26°28′	−6° 6′	.....	+10°48′	−0°55′	−14°19′	−26°12′

NOTE.—The yield of oleoresin from C4 was so small, after the first and second collections, that not enough volatile oil could be obtained on distillation to fill the 100 mm tube.

From this table it is seen that the rotation in most cases is quite constant throughout the year. The most marked exception is A_2 (*P. heterophylla*). It is evident that some distinct change in the biological activity of this tree has taken place, for while the rotation is reasonably constant during the first half of the year, a steady increase in the levo-character of the oil is apparent during the last half. In the case of C_1 (likewise *P. heterophylla*) some-

what the reverse has taken place. A rather marked decrease in the levo-rotation is shown just at the middle of the year, then the rotation remains practically constant during the last half. In the case of C_3, another type of change is represented, the levo-rotation decreasig up to the middle of the season and again increasing during the latter half.

With the limited facts at hand, it is impossible to interpret the significance of these changes. That tree which shows the most marked variation, A_2, is a healthy, vigorous tree, from which variations would be least expected. Nor can an explanation be offered for the wide variations in the optical rotation of oils from the same spieces. All of the trees in Series A are located within 20 yards of each other and have, therefore. the same general conditions of climate, light and soil. Fractionation of the volatile oils from these show practically the same rise in boiling-point for the same volume of distillate. It would seem, therefore, that these volatile oils, consisting so largely of pinene, are mixtures principally of dextro- and levo-pinene, the preponderance of the one or the other determining the optical rotation.

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THE CHARACTER OF THE COMPOUND FORMED BY THE ADDITION OF AMMONIA TO ETHYL-PHOSPHO-PLATINO-CHDORIDE*

BY CHAS. H. HERTY AND R. O. E. DAVIS

By heating together phosphorus pentachloride and spongy platinum, Baudrimont[1] obtained the phospho-platino-chloride $PtCl_2.PCl_3$. Later Schutzenberger[2] prepared the compound $PtCl_2.2PCl_3$ by treating Baudrimont's salt with phosphorus trichloride and he studied the various derivatives of these two substances.

The apparent analogy of these compounds to those of platinous chloride with ammonia led one of us (Herty) in 1901 to investigate them further by physico-chemical methods, in order to determine whether the analogy was real and therefore whether they conformed to Werner's[3] extension of the valence hypothesis. If so, various possibilities of isomerism at once suggested themselves.

These views, in abstract form, were presented to the committee in charge of the C. M. Warren Research Fund and a grant was made for the purchase of platinum. Work was begun at once, but unfortunately a call to another field made impossible the completion of the investigation. The platinum was recovered, sold, and the grant returned.

[1] *Ann. chim. phys.* [4], 2, 47.
[2] *Bull. soc. chim.* [2], 17, 482; 18, 101, 148.
[3] *Z. anorg. Chim.*, 3, 267.
[4] *Ibid.*, 37, 394; 43, 34.

*Reprinted from the Journal of the American Chemical Society, Vol. 30, p. 1084. 1908.

Later, Rosenheim[4] published the results of an investigation covering practically the same ground. He found that the analogy was real and succeeded in obtaining numerous isomers. With the stable ethoxy derivatives, molecular weight determinations, both ebullioscopic and cryoscopic, showed that while the formula of the 1 : 2 compound is normal, that of the 1 : 1 compound must be doubled, thus

$$PtCl_2.2P(OC_2H_5)_3 \text{ and } (PtCl_2.P(OC_2H_5)_3)_2.$$

These facts show both compounds in strict accord with Werner's coordination ideas, namely, that the coordination number of platinum in platinous compounds is four. Accordingly, their formulas would be

$$\left\{ Pt \begin{matrix} Cl_2 \\ [P(OC_2H_5)_3]_n \end{matrix} \right\} \text{ and } \left\{ \begin{matrix} Pt & Cl_2 \\ | & P(OC_2H_5)_3 \\ Pt & Cl_2 \\ & P(OC_2H_5)_3 \end{matrix} \right\}$$

The addition of one molecule of aniline to the former, results in one chlorine atom becoming ionizable, but from the latter, Rosenheim succeeded in obtaining two isomeric substances, a white and yellow modification, each having the formula

$$\left\{ Pt \begin{matrix} Cl_2 \\ P(OC_2H_5)_3 \\ C_6H_5NH_2 \end{matrix} \right\}$$

again conforming to Werner's views.

However, when gaseous ammonia was used instead of aniline an unexpected result was obtained. Two molecules of the base were added for each atom of platinum present, the empirical formula being $PtCl_2.P(OC_2H_5)_3.2NH_3$. According to Werner's views, such a compound should be diionic, as represented by the formula

$$\left\{ \begin{matrix} Cl \\ Pt(NH_3)_2 \\ P(OC_2H_5)_3 \end{matrix} \right\} Cl.$$

But Rosenheim found that silver nitrate precipitated at once both chlorine atoms, even at 0°, and that the molecular conductivity at 25° was

v	32	64	128	256	512
μ	155.9	160.8	160.4	160	162.3

From these facts and from the composition of the double salt with chlorplatinic acid, he concluded that the formula may be

$$\left\{ \begin{matrix} (NH_3)_2 \\ Pt \\ P(OC_2H_5)_3 \end{matrix} \right\} Cl_2,$$

but since such a formula is not in accord with the coordination number of platinous platinum, and since the compound is derived from the double molecule

$$\left\{ \begin{matrix} Cl_2 \\ Pt \\ P(OC_2H_5)_3 \end{matrix} \right\}_2$$

Rosenheim assigned to it the formula

$$\left\{ \begin{matrix} (NH_3)_2 \\ Pt \\ P(OC_2H_5)_3 \end{matrix} \right\}_2 Cl_4.$$

Such a formula appeared to us to be a strained interpretation of Werner's views. Furthermore, the molecular conductivity, as given by Rosenheim, is abnormal in every way. It seemed desirable, therefore, to repeat the preparation of the substance and to study its properties further. Experience gained in the study of the molecular conductivity of complex ammonia compounds at 0°[1]

1 Werner and Herty, Z. *phys. Chem.* vol. 38, p.331.

justified the hope that such a study of this compound might throw more light upon its constitution. Following the directions of Schutenzberger and of Rosenheim, pure spongy platinum was heated with phosphorus pentachloride, the latter freed from trichloride and oxychloride by heating in a current of dry air at 110°. The fused mass on treatment with hot benzene, free from thiophene and purified by freezing, yielded on cooling an abundant crop of well crystallized

$$\left\{ \begin{matrix} Cl_2 \\ Pt \\ PCl_3 \end{matrix} \right\}$$

Freed from benzene, at the same time carefully protected from the action of moisture, the compound wos immediately treated with absolute alcohol in order to convert it into the ethoxy derivative

$$\left\{ \begin{matrix} Cl_2 \\ Pt \\ P(OC_2H_5)_3 \end{matrix} \right\}_2.$$

The alcoholic solution was then placed in a desiccator over two vessels, one containing concentrated sulphuric acid, the other powdered lime, and left to evaporate to crystallization. Removal to a new building necessitated cessation of the work for several months. On resuming, it was found that the solution in the desiccator had evaporated to dryness, no distinct crystallization being noticeable. This mass was dissolved in pure benzene and into the solution dry ammonia gas was conducted. The absorption of ammonia was accompanied by a marked elevation of temperature, the original yellowish tint of the solution gradually faded and then, rather suddenly, a mass of white crystals separated, the mass becoming almost solid. The completion of the reaction was indicated by the return to normal temperature. The crystal broth was set aside and owing to the exigiencies of other work, two weeks elapsed before the crystals were separated from their mother liquor. The substance, freed from benzene, was recrystallized from alcohol and obtained in a very pure form.

A portion of the substance dissolved in water showed no acid reaction, although Rosenheim found an immediate acid reaction and explained the peculiar results he obtained from a study of its molecular conductivity by assuming a rapid hydrolysis of the compound. Analysis showed:

			Found by Rosenheim	
	I	II	I	II
Cl (Ionizable).....	8.02	7.96	15.08	14.42
Pt.....	42.30	41.40	41.68	.....
NH_3	7.04	7.18	7.19	.....

	Theoretical for	
	$\left[Pt\begin{matrix}(NH_3)_2\\P(OC_2H_5)_3\end{matrix}\right]_2Cl_4$	$\left[Pt\begin{matrix}Cl\\(NH_3)_2\\P(OC_2H_5)_3\end{matrix}\right]Cl$
Cl (Ionizable).....	15.23	7.62
Pt.....	41.82	41.82
NH_3	7.30	7.30

Ionizable chlorine was determined at room temperature by precipitation of the water solution of the substance with excess of silver nitrate. The filtrates remained clear even after standing several weeks. Platinum was determined by Rosenheim's method[1], ammonia by the Kjeldahl method.

Effort was then made to determine the total chlorine by Rosenheim's method[1] but concordant results could not be obtained. Determinatians of the total chlorine by Stepanow's[2] method gave:

Total Chlorine		
I.	II.	Theoretical
15.19	15.00	15.23

Determination of the molecular conductivity at 25° showed

v	32	64	128	256	512	1024	2048
μ.....	95.79	100.13	106.09	113.49	119.70	127.97	138.98
Found by Rosenheim	155.9	160.8	160.4	160.0	162.3	.....	.....

1 *Z. anorg. Chem.*, Vol. 37, p. 385.
2 *Ber.*, Vol. 39, p. 4056.

The values for μ found by us agree closely with the figures obtained by Werner and Miolati[3] and by Werner and Herty[4] for all diionic complex ammonia compounds. No evidence of hydrolysis could be detected in the solution which had been used for the determination of the molecular conductivity, the reaction was perfectly neutral.

From all the above facts, it is evident that the formula of our compound is

$$\left\{\begin{matrix} Cl \\ Pt(NH_3)_2 \\ P(OC_2H_5)_3 \end{matrix}\right\}Cl,$$

a formula strictly in accord with Werner's coordination law. It would seem further that we have here another case of isomerism of inorganic compounds.

In order to gain further knhwledge of the conditions which determine the formation of the one or the other of these substances, new experiments were begun. A fresh portion of the compound

$$\left\{\begin{matrix} Cl_2 \\ Pt \\ P(OC_2H_5)_3 \end{matrix}\right\}_2$$

was prepared. Twenty-one grams of the substance were dissolved in seventy-five cc. of benzene and the solution was divided into three equal portions. In one, designated A, the substance was prepared under the conditions above described. In another, designated B, the crystals were promptly separated from the benzene liquor. In the third portion, designated C, the temperature was maintained at 6° throughout the experiment.

In order to insure as far as practicable a uniform addition of ammonia in the several experiments, thirty-five grams each of finely pulverized lime and ammonium chloride were thoroughly mixed and placed in a 500 cc. round bottom flask and heated in a

[3] *Z. phys. Chem.*, Vol. 12, p. 35; Vol. 14, p. 506; Vol. 21, p. 225.
[4] *Ibid.*, Vol. 38, p. 331.

bath of cottonseed oil. At 220° copious evolution of ammonia began. The gas, dried over quicklime, was passed through the benzene solution of the ethoxy compound for forty-five minutes, during which time the temperature of the oil bath was gradually raised to 245°.

The benzene solution was placed in a 50 cc. round bottom flask provided with a three-hole rubber stopper through which passed a thermometer dipping into the solution and the inlet and outlet tubes for the ammonia.

Experiment A: The initial temperature of the benzene solution was 22°. When the temperature of the oil bath surrounding the generator reached 220° the temperature in the absorption flask began to rise. After five minutes it was 25°, after ten minutes 33°, after fifteen minutes, 38°. Meanwhile the yellow color of the original solution gradually faded. After twenty minutes the temperature was 40°, then suddenly the separation of the white substance took place to such an extent that the mass became almost solid. A portion of the substance was at once removed from the absorption flask, pressed between folds of drying paper and dissolved in water. No evidence of hydrolysis could be detected, the solution being perfectly neutral to indicators, As Rosenheim found that his salt was strongly hydrolyzed, it was decided to continue the passage of the ammonia gas into the crystal broth longer. Accordingly, this was continued for twenty-five minutes more, the temperature of the solution falling gradually. The vessel tightly corked was allowed to stand two weeks. At the end of this time the crystals were filtered from the benzene and pressed between folds of drying paper, then recrystallized from absolute alcohol and labeled "A".

Experiment B: This was a repetition of Experiment A except that the crystals were removed from the benzene immediately after the conclusion of the experiment. The maximum temperature observed in the absorption flask was 44°. The separation of the crystals took place twenty-two minutes after the temperature of the oil bath surrounding the ammonia generator had reached 220°. The substance recrystallized from alcohol was labeled "B".

Experiment C: This was a repetition of Experiment B except that the temperature of the benzene solution was kept constantly at 6°. The crystal separation took place twenty-four minutes from the time the oil bath reached 220°. Recrystallized from alcohol the substance was labeled "C".

The results of the analyses of the three preparations follow:

	Ionizable chlorine		Total Chlorine	Theoretical chlorine for $\left[\begin{matrix}Cl\\Pt(NH_3)_2\\P(OC_2H_5)_3\end{matrix}\right]Cl$.	
	I	II		Ionizable	Total
A	7.66	7.64	15.16	7.62	15.23
B	7.74	7.66	15.17	.....	.....
C	7.47	7.49	15.01	.....	.....

From these results it is evident that we have succeeded in preparing only the normal salt

$$\left\{\begin{matrix}Cl\\Pt(NH_3)_2\\P(OC_2H_5)_3\end{matrix}\right\}Cl.$$

And yet Rosenheim's directions have been faithfully followed and his description of the absorption experiment coincides with our observations. It is therefore not considered profitable to further undertake the preparation of Rosenheim's compound until more specific directions are given. Meanwhile, it is our intention to try to prepare the compound

$$\left\{\begin{matrix}Cl_2\\Pt\,NH_3\\P(OC_2H_5)_3\end{matrix}\right\}$$

analogous to the aniline compound prepared by Rosenheim, and to gain further light upon the character of the reaction by which the addition of ammonia changes the non-ionizable compound

$$\left\{\begin{matrix}Cl_2\\Pt\\P(OC_2H_5)_3\end{matrix}\right\}.$$

into the ionizable compound

$$\left\{\begin{matrix} Cl \\ Pt(NH_3)_2 \\ P(OC_2H_5)_3 \end{matrix}\right\}Cl.$$

UNIVERSITY OF NORTH CAROLINA
CHAPEL HILL, N. C.
March 11, 1908

THE VOLATILE OIL OF PINUS SEROTINA*

BY CHAS. H. HERTY AND W. S. DICKSON

Scattered among the forests of Long Leaf pine along the Atlantic seaboard, there are found, usually in mixed stands, patches of Pond pine (*Pinus serotina*) and Loblolly pine (*Pinus taeda*). These pines are seldom subjected to turpentining, as the yield of oleoresin is not so plentiful as from the predominating types *Pinus palustris* and *Pinus heterophylla*. Nor are the two species usually distinguished locally, the name "black pine" being applied to each. The striking odor of the wood of *Pinus serotina* when freshly cut made desirable an investigation of its volatile oil, and in collaboration with the U. S. Forest Service, the oil has been studied in this laboratory during the past year. Well identified trees were selected in Florida. The trees were regularly chipped throughout one season of eight months. The product from each tree was collected every eight weeks. The oleoresin closely resembles that from Cuban pine (*P. heterophylla*) being quite liquid and containing relatively about the same proportion of crystalline acids. To this low percentage of crystalline matter is to be assigned doubtless, as in the case of *P. heterophylla*, the absence of "scrape" formation on the scarified surface of the tree, a formation so typical of *P. palustris*).

The volatile oil was distilled from the oleoresin by steam in the apparatus described on page 865 above. The oleoresin evidently contains a greater portion of mucilaginous substances than that from the more common pines, for it was much more difficult to distil. On heating to 140°, the usual temperature of distillation, and introducing steam, the easily molten mass froths badly. This could be avoided only by raising the temperature at the out-

*Reprinted from the Journal of the American Chemical Society. Vol. 30, p. 872. May, 1908.

set to 160°. At this temperature, the viscosity is diminished sufficiently to enable a complete distillation to be carried out without frothing. During the latter part of the summer, however, and during the autumn, the amount of this mucilaginous substance evidently increased, and to such an extent that it became practically impossible to distil off the volatile oil. Partial success was secured by he addition of concentrated sodium hydroxide solution to the distilling flask.

The resin left after distillation is pale yellow, similar to the best grades of commercial resin. Acid number 167.

The volatile oil, freed from water by standing in contact with calcium chloride, was a limpid liquid with a fragrant odor suggesting at once the presence of limonene. The physical constants of the oil follow:

Sp. gr.: 20°, 0.8478.
Sp. rotation: 20°, —105°36.
Index of refraction: 20°, 1.47$\frac{3}{4}$4.
Acid number: 0.
Saponification number: 1.54.
Iodine number: 378.
Solubility in ethyl alcohol at 22.5°:
95 per cent. alcohol 1.35 parts required to dissolve 1 part of volatile oil.
90 per cent. alcohol 4.80 parts required to dissolve 1 part of volatile oil.
85 per cent. alcohol 8.10 parts required to dissolve 1 part of volatile oil.
80 per cent. alcohol 16.20 parts required to dissolve 1 part of volatile oil.
70 per cent. alcohol 56.00 parts required to dissolve 1 part of volatile oil.

Comparative evaporation with the volatile oil of *P. palustris*, at room temperature, in shallow watch glasses, 0.2 gram of each used.

Time	P. palustris Per cent.	P. serotina Per cent.
Loss after ½ hour	35.7	20.30
Loss after 1 hour	62.5	37.30
Loss after 1½ hours	91.7	53.40
Loss after 2 hours	96.0	68.47
Loss after 5 hours	97.8	98.80

On fractionation the following results were obtained:

Temperature	Per cent. distillate	Index of refraction, 20°	Rotation in 100 mm. tube 20°
172-175°	27.4	1.4716	—87°53'
175-180°	57.0	1.4724	—92°21'
180-185°	8.4	1.4744	—92°14'
185-+	7.2	1.5045	.....

Repeated fractionation at atmospheric pressure showed some polymerization. From a fraction, 175-176°, a large yield of limonene tetrabromide was obtained. Melting point 103°-103°. The solution of the tetrabromide in chloroform was levo-rotatory, — 70-0°.

A study of the oxygen absorbing power of this volatile oil in comparison with that of the ordinary spirits of turpentine obtained from *P. palustris* showed a much larger absorption by the oil of *P. serotina* during the early days of the experiment, but the total absorption after three months' exposure to northern light was practically the same in each.

These results show a wide variation in the optical rotation of the volatile oils from the individual trees, even among trees of the same species. In a general way the figures give support to Long's view, namely that the volatile oils from the *Pinus palustris* are dextro-rotatory and those from *Pinus heterophylla* levo-rotatory. That this is not strictly true, however, is evidenced by the dextro-rotation of A_2 (*P. heterphylla*) and more especially by the levo-rotation of C_3 (*P. palustris*).

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MICROPEGMATITE AT CHAPEL HILL.

BY H. N. EATON.

During the course of an investigation of the Chapel Hill granites now in progress an occurrence of micropegmatite was discovered which seems worthy of special mention. The binary granites were found to contain micrographic intergrowths of quartz and feldspar. The one showing the best development of this phenomenon is described below.

This rock occurs in a slightly weathered condition just beyond the village limits on the slope of the hill along the Hillsboro road and south of Bolin's Creek. Its extent and relation to the other rocks of the igneous complex of the region are unknown.

In handspecimen, the rock is grayish pink in color, and fine grained. No hint of the arrangement of the minerals is given from a freshly broken surface owing the uniform fineness of grain.

In thin section, the mineral content is seen to comprise plagioclase, orthoclase, microcline, and some accessory magnetite. The striking feature of the slide is the arrangement of the quartz and alkali feldspar in the micropegmatitic relation.

With the exception of the magnetite, plagioclase was the first mineral to crystallize. It occurs sparingly in short, stout prisms, for the most part idiomorphic, but in places showing absorption by the later formed minerals. Twinning occurs after both the carlsbad and albite laws. Owing to strong kaolinization the albite striations are frequently difficult to recognize. The maximum extinction angle noted was 13 degrees, thus placing the species between oligoclase and andesine, and nearer the latter of the two.

Quartz and alkali feldspar occur mutually inclosing and interpenetrating each other in micrographic intergrowths. The

quartz is best seen in this relation in long, spindle-shaped forms with parallel orientiation over a considerable portion of the field. These are wholly inclosed in orthoclase or microcline and extinguish simultaneously over wide areas. Measurements with a micrometer eye-piece gave lengths of .58 m.m., .62 m.m., and .69 m.m. for the longer quartz spindles. In places the large spindles radiate from centers in which clusters of smaller quartz crystals are imbedded in orthoclase and microcline. There are a few places where quartz occurs in the triangular and knee-shaped form characteristic of graphic granite as originally described.

The orthoclase is very abundant and forms a large percentage of the rock. Crystal boundaries are very indistinct. Large areas inclosing the quartz spindles extinguish as single crystals separately from the quartz.

Microcline is less abundant than orthoclase, and the areas occupied are smaller. Crystal boundaries are indistinct.

The impression first given by the slide is that the orthoclase and microcline were the last to crystallize, and constitute a groundmass in which the quartz is set, but a careful examination reveals many quartz-feldspar boundaries in which the two minerals are mutually interpenetrating. Many quartzes also contain small poikilitic inclusions of orthoclase. It is therefore probable that the quartz and the alkali feldspars crystallized contemporaneously.

UNIVERSITY OF NORTH CAROLINA
CHAPEL HILL, N. C.
March 11, 1908

ABSTRACTS

ON THE EFFECT OF COMPLETE ANEMIA OF THE CENTRAL NERVOUS SYSTEM IN DOGS RESUSCITATED AFTER RELATIVE DEATH. D. H. Dolley and George Crile, M. D.[1] Jour. of Expe. Med., vol. 10, Nov. 1908.

This study of brain anemia is the sequence of provious work on the resuscitation of animals killed by anaesthetics and asphyxia,[2] which may be briefly summarized as follows: By measn of a centripetal arterial infusion of salt solution under sufficient pressure, together with the injection of one to two cubic centimeters of 1-1,000 adrenalin chloride in mass dose early and along with the infusion, and the simultaneous employment of vigorous artificial respiration and gentle but firm cardiac massage through the unopened thorax, a heart which has ceased to beat may be excited to resume beating within certain limits. Up to five minutes of total cessation of function, these efforts are uniformly and readily successful, provided that the full technique has been used: up to ten minutes, there is an occasional failure, but after that the chances of success become progressively less. Our limit was five minutes in puppies (three cases).

To determine the limits of recovery after the total anemia of the central nervous system incident to a state of relative death, a series of thirty dogs was killed by chloroform and resuscitated after varying times from three to fourteen minutes. Under five minutes the recovery of function was rapid and strikingly free from the after effects which characterized longer periods. Of seven animals between the periods of five minutes and six and one half

[1]From the Laboratory of Surgical Physiology, Western Reserve University, and the Pathological Laboratory, University of North Carolina.

[2]*Jour. of Exper. Med.*, vol. 18, p. 718, 1906.

minutes, only one died apparently as a direct result of the anemia, but of twelve between the periods of seven minutes and eight and one half minutes, only one, after seven and one half minutes, recovered. The remaining dogs all died.

After a resuscitation, the course of events in the animals which succumbed, while limited according to the extent of the reanimation, was similar to that in the dogs which did eventually recover. Many of the dogs showed more than a mere reflex revival there being some temporary manifestation of special senses and higher faculties in addition. In general, three stages were to be observed. A state of hyperexcitability followed reanimation, reaching its maximum in one to three hours, when retrogression began. This second stage was characterized by the onset of uncontrolled muscular movements, either coordinate or convulsive, lasted a longer time, and gradually passed into the third stage of depression and paralysis. The crisis in practically all the experiments was reached in from twelve to twenty-four hours. Then death quickly ensued or distinct improvement of nervous functions shortly began, continuing more or less rapidly till complete restoration, though the convalescent period lasted in two dogs four and six weeks respectively. There was no intermediate condition of fatal outcome delayed for several days except in several cases in which death was due to accidental organic lesion. Up to a certain point, not to be exactly limited, but roughly six minutes, the after effects were not marked, and the second, third, or fourth day brought complete recovery. Beyond the six minute limit, however, there was a great deal of after effect, increasing out of all proportion to the increase in the duration of the period of anemia, reaching as well in the dogs which finally recovered a temporary state in which the animal was little more than a cardio-respiratory mechanism.

(The sequence of return of the various functions and reflexes and the special phenomena following a resuscitation are discussed in detail.)

Histological examination both of presumptive recoveries and fatal cases was made by ordinary methods and those of Nissl and Marchi. The neurocytes of the fatal cases uniformly presented the greatest change, not merely chromolytic but here and there

indicative of cell death. Marchi's method further supported these findings by proving the existence of fibre degeneration. Finally, showing the narrowness of the escape, the best result in recovery, seven and one half minutes in time, which at the end of four weeks had apparently entirely returned to a normal state, by the Marchi method had a degeneration of a number of fibres localized in the pyramidal fasciculi, which were traced from the cord to the cortex, and in Flechsig's fasciculus, as well as a more sparsely scattered degeneration of both ascending and descending fibres elsewhere. The results of the histological examination place the limits of experimental resuscitation upon an anatomical basis.

CONCLUSIONS

1. In dogs anesthetized by ether for preparation and killed quickly by chloroform, the average limit of total cerebral anemia, estimated from the cessation of the heart sounds to the return of circulation, which admits of recovery, is between six and seven minutes. Any recovery beyond seven and half minutes would be exceptional, and the ulterior limit appears to be under ten minutes, hitherto stated as the most conservative figure after other modes of investigation.

2. Further, experience with resuscitation of animals killed by anesthetics and asphyxia, embracing numerous unrecorded experiments as well as those forming the basis of the present article, establishes our former conclusion, that the procedures detailed afford a reliable method within its limitations, and certainly uniformly successful within the limits compatible with the recovery of the central nervous system.

BINDERS FOR COAL BRIQUETS, by J. E. Mills. Bulletin 343 of the United States Geological Survey.

The bulletin contains a report of investigations made at the Fuel-Testing Plant, St. Louis, Mo., by the author. The characteristics of good briquets are discussed and the general conditions governing the use of binders. A very large number of different binders were investigated in the laboratory, the effort having been made to include in the list all binders which it was thought

might be used commercially in the United States, as well as certain other substances which seemed fitted to throw light on the laws governing the action of the binder. Attempt was also made to study such modifications and combinations of the different binders as it seemed might produce more efficient commercial results. The author gives the following summary of the investigations:

"Definite answer to the question 'What is the best binder to use in making briquets?' depends, as repeatedly emphasized in this paper on the locality, on the character of the coal, and on the purpose for which the briquets are intended. For purposes of a brief comparison consideration is given to the binders available for a coal which is fairly easy to briquet and which cakes rather readily. A few coals will briquet with somewhat less and others require greater percentages of binder, but an endeavor has been made in the following summary to strike a reasonable average.

"The experiments herein reported show that, in general, for plants situated where it can be obtained, the cheapest binder will prove to be the heavy residuum from petroleum, often known to the trade as asphalt. Four per cent of this binder being sufficient, its cost ranges from 45 to 60 cents per ton of briquets produced. This binder is particularly available in California, Texas, and adjacent territory.

"Second in order of importance comes water-gas tar pitch. Five to six per cent usually proving sufficient, the cost of this binder ranges from 50 to 60 cents per ton of briquets produced. As water-gas pitch is also derived from petroleum, it will be available more particularly in oil-producing regions.

"Third in order of importance is coal-tar pitch. Being derived from coal, this binder is very widely available. From 6.5 to 8 per cent will usually be required, and the cost ranges from 65 to 90 cents per ton of briquets produced.

"Of local importance, where the price permits, are natural asphalts and tars derived from wood distillation. The price of each of these binders varies greatly with the locality, but there are doubtless places where they could compete with the binders

above mentioned. Wax tailings could be used with an easily caking coal.

"Pitch made from producer-gas tar is not yet on the market, but it will produce excellent briquets with a lower percentage of binder than other coal-tar pitches. It will doubtless be available in the future.

"Briquets excellent in all respects except that they are not waterproof can be made by using 1 per cent of starch as a binder, the cost of which is 20 cents per ton of briquets produced. Extra care is necessary in drying and handling these briquets, and this adds to their cost.

"The waste sulphite liquor from paper mills also producees excellent briquets except that they are not waterproof. At present it is a troublesome waste product dissolved in much water. Its utilization for this purpose will bear further investigation.

"Of inorganic binders, magnesia might be utilized, as its probable cost would not exceed 22 to 30 cents per ton of briquets produced. Other inorganic binders, while available as regards price, would not make first-class briquets.

"The briquetting of lignite coal offers a peculiarly difficult problem. If the lignite cakes in the fire, asphaltic residues from petroleum or water-gas tar pitch may be used as binder, larger percentages being required than for ordinary coals. The most promising binders for lignites that do not cake are starch, sulphite liquor, and magnesia. Lignites may be briquetted without binder if they are to be burned on grates specially constructed to overcome the tendency to fall to pieces in the fire.

"Attention is called to the suggested method of deciding as to the value of coal-tar pitch for briquetting purposes. The method is likewise applicable to asphalts and petroleum residues generally; (1) The pitch or tar is distilled and all oils coming off below 270° C. are rejected as being of no value; (2) the flowing point of the portion to be used in briquetting is determined (this should generally not be less than 70° C.) (3) the pitch is extracted with carbon disulphide. The smaller the amount of residual carbon the more satisfactory is the pitch. The less readily the coal cakes the higher must be the flowing point of the pitch. If a pitch cracker is used, the pitch to work successfully

on a hot summer's day must have a flowing point above 120° C. In the winter pitch with a flowing point of 100° C. may be used. All softer pitches and asphalts have to be melted and mixed in liquid form with the coal.

A pitch with a very high softening point, above 150° C., should be either thinned or superheated in the mixer. The efficient use of a binder depends very largely on the proper regulation of the conditions in the mixer. The presence of low-volatile compounds in the pitch to be used as a binder increases the smoke in burning; and also increases the tendency of the briquet to soften and crack open in advance of combustion, owing to the volatilization and escape of these compounds.

"The main problem in briquetting is to find a suitable binding material at sufficiently low cost. When the difference in price between the slack coal and the first-class lump coal is $1, the cost of briquetting should not exceed this amount. Of this the binder must cost less than 60 cents per ton, as the cost of manufacture averages about 40 cents. To leave out of consideration the possible advantages in the use of briquetted coal over run-of-mine coal, due to the greater efficiency and smokelessness of briquets, it will probably not be necessary to pay any attention to binding materials costing $1.25 or more per ton of briquets produced."

THE VOLATILE OIL OF PINUS SEROTINA, by Chas. H. Herty and W. S. Dickson. Jour. Am. Chem. Soc., vol. 30, p. 872. May 1908.

A study of the volatile oil obtained by distillation with steam from the oleo-resin of Pinus Serotina (Pond Pine) showed that it contained large quantities of limonene instead of the more common pinene present in ordinary spirits of turpentine. The oil showed the following physical contents:

Specific Gravity: 20° 0.8478
Specific Rotation: 20° —105° 36'
Index of Refraction: 20° 1.4734
Acid Number: 0
Saponification Number: 1.54
Iodine Number: 378
On Fractionation the oil showed:

Temperatures	Per Cent of Distillate
172-175	27.4
175-180	57.0
180-185	8.4
185-+	7.2

The limoene was identified by conversion into the tetrabromide.

THE CHARACTER OF THE COMPOUND FORMED BY THE ADDITION OF AMMONIA TO ETHYL-PHOSPHO-PLATINO CHLORIDE, by Chas. H. Herty and R. O. E. Davis. Jour. Am. Chem. Soc., vol. 30, p. 1084. July 1908.

Efforts to prepare Rosenheim's Salt $\left\{ \begin{matrix} (NH_3)_2 \\ Pt \\ P(OC_2H_5)_3 \end{matrix} \right\}_2 Cl_4$ failed. On each attempt, following closely Rosenheim's directions, the salt $\left\{ \begin{matrix} Cl \\ Pt\ (NH_3)_2 \\ P(OC_2H_5)_3 \end{matrix} \right\} Cl$ was obtained. Only half of the chlorine in this compound can be precipitated by silver nitrate. Determinations of its molecular conductivity show close agreement with analagous di-ionic complex ammonia compounds studied by Werner, Miolati and Herty. The character of the compound agrees fully with the co-ordination hypothesis of Werner.

CORROSION OF IRON, by R. O. E. Davis. Ch. Eng., vol. 5, p. 174. 1908.

The corrosion of iron in explained by different workers in several different ways, but the main discussion is whether it is oxygen or carbon dioxide that is essential to rusting. Experiments were devised to determine this point, from which the conclusion is drawn that corrosion of iron takes place with water and oxygen but not with water and carbon dioxide. The presence of carbon dioxide may accelerate the reaction but is not essential to it.

THE OPTICAL ROTATION OF SPIRITS OF TURPENTINE, by Chas. H. Herty. Jour. Am Chem. Soc., vol. 30, p. 863. May 1908.

The results of this investigation were obtained from oleo-resins from individual trees in Florida of Pinus Palustris and Pinus Heter-

rophylla. The collections of the oleo-resins from which the volatile oils were distilled were made at regular intervals throughout a complete season, March to November. A number of trees of each species were used.

The optical rotation of the severel volatile oils obtained on distillation showed a marked variation. Those from Pinus Palustris were generally dextrorotatory, varying however from +1° 23' to +18° 18' (100 mm. tube). One specimen showed a levorotation of —7° 26'. The oils from Pinus Heterophylla were levorotatory, with one exception +0° 15', varying from —7° 31' to —29° 26'.

While these wide variations were noticed in the specimens from the individual trees, there was but slight variation in the specimens from the same tree throughout the season.

Vanishing Point